Sampling and Analysis Plan for the 2008 Sediment Quality Evaluation of Grand Lake

October 2008

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1.0 Introduction

1.1 Background

Located in northeast Oklahoma, the Grand Lake O' the Cherokees (Grand Lake) is an artificial lake created by the damming of the Neosho River. Spanning two counties and draining about 10,000 square miles, Grand Lake is the receptor of the Neosho and Spring Rivers, which flow through the historic Tri-State Mining District. Commercial mining in this area extended for more than a century, producing a major share of the world's zinc and lead ores throughout this time period.[1]

The long history of mining in this region has adversely impacted the aquatic and terrestrial ecosystems and potentially affected resident biota. Although the concentration and distribution of mining-related contaminants has been documented in the Neosho and Spring Rivers and tributaries,[2] the status of Grand Lake is not well known. Limited sediment sampling in upper Grand Lake was conducted by the Oklahoma Water Resources Board in 1982 and USGS in 2002 and 2004; the sampling locations are reported in Figure 1, while the results of metals analysis from these sampling events are shown in Table 1. A recent analysis of the Empire Lake (Kansas) sediments suggests that contaminated sediment has washed past the Empire Lake Dam and has been deposited as far as Grand Lake (Juracek 2006). Given the heavy metal contamination and adverse effects documented in the upstream watersheds of the Tri-State District, an assessment of the current status of Grand Lake is warranted and necessary to fully assess injury to Trust resources in Oklahoma.

In order to assess the impact to Grand Lake and fill current data gaps, sediment samples will be collected from select sites in the lake and analyzed for metal contamination in two phases. The Phase I sampling will take place in early October 2008 and will focus mainly on the lower two thirds of Grand Lake, the Route 59 bridge to Pensacola Dam. The Phase II sampling will take place October 20th - 24th and the focus will be lake wide from the confluence of the Spring and Neosho Rivers to the Pensacola Dam.

This document, termed the Sampling and Analysis Plan (SAP), includes the Field Sampling and Quality Assurance Plan (FSP/QAPP) and describes the standard procedures (SOPs) that will be used to collect and analyze the Phase I - 96 sediment samples (Tables 1 to 4) from the lower two-thirds of Grand Lake to evaluate contamination by heavy metals by XRF (with confirmatory samples) and use to collect and analyze the 40 - Phase II sediment samples from the entire lake to determine toxicity of the sediments to the amphipod *Hyalella azteca* in standardized 28-d sediment toxicity tests (Tables 5 to 8).

[1] The region was also an important producer of lead, silver, cadmium, germanium, and gallium (Hagni, 1989).
[2] The adverse effects of mining in the Tri-State District have been documented in several sources including, but not limited to: Dames and Moore, 1993 and 1995; Ferrington et al, 1995; USGS, 2005, MacDonald et al. 2008.

1.2 Sampling and Analysis Plan for the Grand Lake

A sampling and analysis plan (SAP) consists of three primary elements, including a quality assurance project plan, a field sampling plan, and an associated health and safety plan (HSP). The QAPP describes the policy, organization, functional activities, quality assurance and quality control protocols necessary to achieve project data quality objectives (DQOs) dictated by the intended use of the data, while the FSP provides guidance for all fieldwork by defining in detail the sampling and data gathering methods to be used on the project.

This document includes the essential elements of both an FSP and a QAPP. Development of a separate QAPP document will be considered if the investigation proceeds beyond this preliminary sampling effort. The HSP will be developed separately.

2.0 Sampling Objectives

The 2008 field sampling program is intended to provide the information needed to determine if discharges from historic mining operations are likely to have degraded sediment quality conditions in the Grand Lake. More specifically, the objectives of the sampling program are to:

Phase I:

1) Obtain data on the concentrations of target metals in 96 sediment samples collected from the lower two-thirds of the lake [i.e., analysis of dried sediment samples using a portable X-ray fluorescence (XRF) for metals as described in EPA Method 6200.]

2) Confirm the results of XRF-based chemical analyses of sediment samples through analysis of a sub-set of the collected samples for target metals. Samples will be sieved to 64 µm, digested following EPA Method 3052, and analyzed by Inductively Coupled Plasma/Atomic Emission Spectrometry (ICP-AES, EPA Method 6010B).

Phase II:

Determine the toxicity and bioavailability of metals to sediment-dwelling organisms a in the lake. The Phase II sampling locations will be dictated by the results of the Phase I XRF results. The Phase II collection will include about 10 samples with low metals chemistry, 10 samples with moderate metals chemistry, and 20 samples with high metals chemistry (based on sediment quality guidelines established by USGS as sensitive indicators of metal bioavailability in contaminated sediments of the TSMD)

3.0 Sampling Program Design

Phase I:

Sediment sampling will take place in early October over the course of three to four days, with a reserve day set aside in case of inclement weather or other extenuating circumstances. Three sampling teams, each with three people, will collect approximately 96 sediment samples at pre-designated sampling sites. Sample sites will be chosen along

seven transects that span the Grand Lake study area. Each sample will be analyzed using XRF technology, as indicated in Section 7.0. A subset of samples will be corroborated through laboratory analysis. All attempts will be made to conform to the methodology recently employed by EPA during sediment sampling of the Spring River, to allow for comparison across sites.

Phase II:

Toxicity testing of sediments associated with Phase II is scheduled October 20 -24 2008. The results of the XRF metal analyses on the Phase I sediment samples will be used to select about 40 sediment samples for toxicity testing by CERC. The distribution of Phase II sediment samples will include about 10 samples with low metals chemistry, 10 samples with moderate metals chemistry, and 20 samples with high metals chemistry (based on sediment quality guidelines established as sensitive indicators of metal bioavailability in contaminated sediments of the TSMD; MacDonald et al. 2008). About 2 L of sediment from each of the 40 Phase II sediment sampling locations will be collected by USFWS and delivered to CERC in Columbia MO. Toxicity of the Phase II sediment samples will be evaluated using 28-d whole-sediment toxicity tests with the amphipod *Hyalella azteca* following standard ASTM (2008a) and USEPA (2000) methods (Tables 5 to 7). To assess genetic strain or life stage sensitivity of test organisms, subsamples of amphipods used in toxicity tests will be evaluated by conducting an acute toxicity test with a reference toxicant (NaCl) following standard test methods (Table 8; USEPA 2000, ASTM 2008a,b). Statistical analyses of the sediment toxicity data and the reference toxicant data toxicity data will be performed in accordance with requirements outlined in USEPA (2000) and ASTM (2008a,b).

4.0 Sampling Locations and Frequency

Phase I

The sampling locations that were selected for inclusion in the 2008 field sampling program for Grand Lake are shown in Figure 1, with coordinates specified in Table 2. Sampling will consist of a series of transects across the lake that will broadly characterize current conditions of the targeted lower two-thirds. Twelve transects have been selected across which eight samples will be collected, for a total of 96 samples (Figure 1).

Sediment samples will be collected at each of the sampling stations identified in Table 2, unless:

- It is not possible to access the predetermined site;
- Inadequate or unusable sediment (i.e., rocks or gravel) is available at the site; or
- Unsafe conditions are apparent at the site.

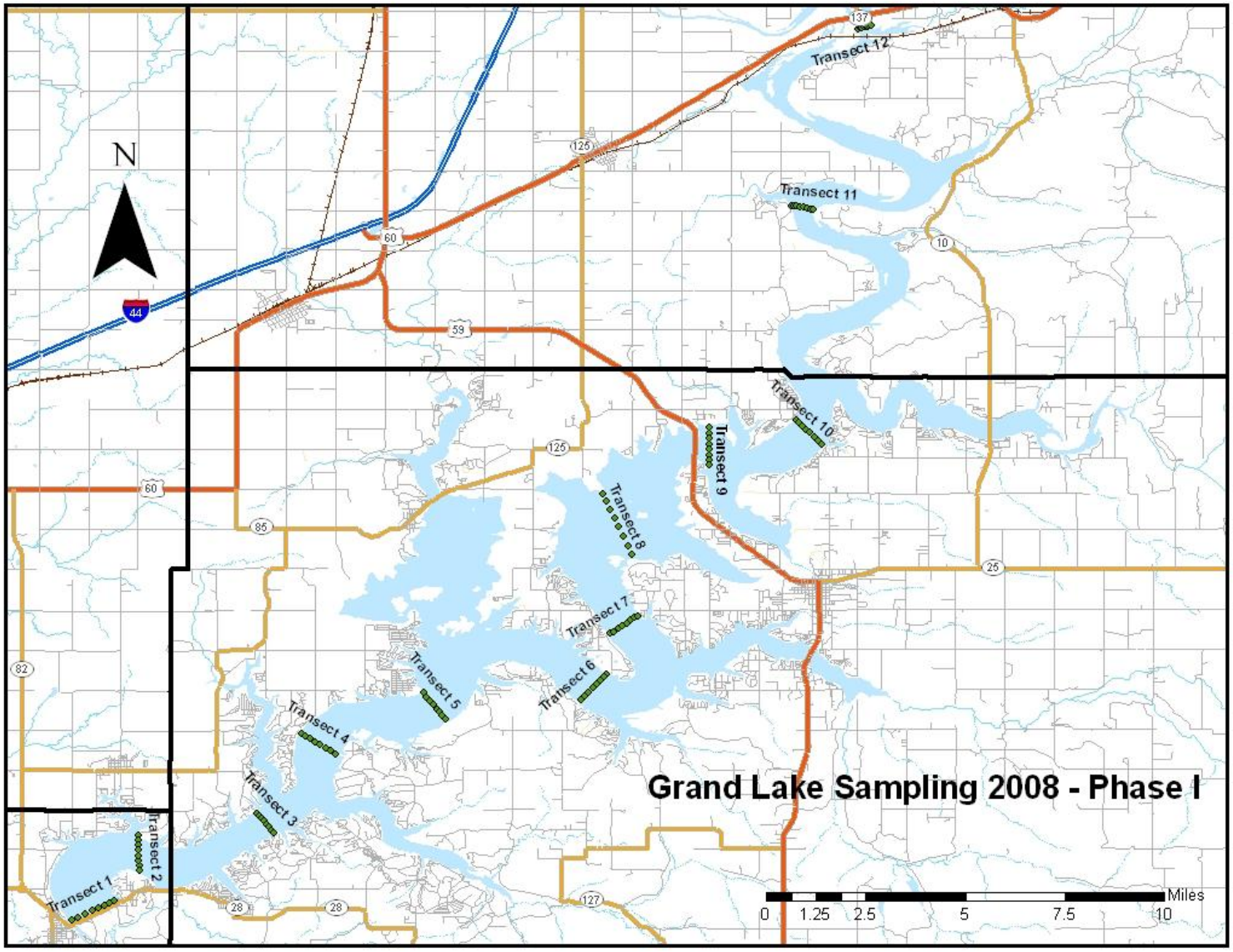

Figure 1 – Sampling locations for 2008 Grand Lake study

If it is not possible to sample surficial sediments at the predetermined site, then the sediment sample may be collected at an alternate site located with 25 m of the original station. The coordinates of the alternate sampling station should be recorded once an acceptable sample has been collected. If an acceptable sample cannot be collected at the adjusted sampling location, the station should be abandoned and no sample will be collected at the station.

Each sampling station will be sampled using sediment grab techniques during the January 2008 sampling program. At each station, a grab sediment sample will be collected and the upper 10 cm of material from the grab will be homogenized to facilitate sub-sampling of the sediment. The target analytes for the surficial sediment samples are shown in Table 4.

Phase II

There is currently insufficient information to direct sediment sampling efforts to areas of suspected contamination or known depositional areas along the lake bottom. Therefore, the sample locations for Phase II will be determined based on the XRF results of Phase I (and previous sampling in 2007).

5.0 Sampling Timing

The sediment sampling to support the Grand Lake sediment quality assessment will be conducted in two phases that will each last four days in October 2008. Sediment samples in Phase I will be collected at a total of 96 sampling stations and Phase II will collect a total of 40 sampling stations in Grand Lake. Because the sampling program will be conducted over a relatively short period of time, there is no need to randomize the schedule for collecting the sediment samples. Phase II sediment samples will be collected October 20-24 for delivery to CERC in Columbia MO by October 24, 2008.

6.0 Sample Designation

The sample numbering system that will be used to designate sediment samples that are collected to support the assessment of sediment quality conditions in Grand Lake is presented in Table 2.

7.0 Sampling Equipment and Procedures

7.1 Information to be Collected at Sampling Stations

The following basic information will be collected and recorded at each sampling station:

- Sample station name and number;
- Sampling date and time;
- Type of vessel used (including length and power);
- Latitude and longitude (i.e., of actual location of sampling, including datum, instrumentation used, and any problems encountered in locating the station);

- Weather conditions, including precipitation, wind speed and direction, lake state (i.e., wave height), etc.;
- Type of sampler used;
- Names of sampling personnel; and
- Water depth.

7.2 Equipment and Sampling Procedures

Table 3 lists the equipment that will be used to collect sediment samples for the 2008 Grand Lake field sampling program. Samples will be collected following guidance provided in ASTM 2008c).

Phase I:

Sediment samples will be collected using a stainless steel Ekman dredge sampler (6 x 6 x 6 inches), targeting no more than the approximate top 10 cm of sediment.

At each sampling station, the sediment sampler will first be rinsed with site water, then with an Alconox soap solution, and finally with site water to prevent cross-contamination among samples. Then, the sampler will be deployed, gently extracted from the sediment, and slowly and carefully raised through the water column to minimize sample disruption during retrieval. Once the sampler has been retrieved from the water and secured on the deck of the boat (in a stainless steel tray), the screen doors will be opened and any excess water that is retained on the sediment surface will be siphoned off (using a thoroughly cleaned siphon or turkey baster). The following information should be recorded following retrieval of the sampler (also see Addendum 1A):

- Unusual events that occurred during sampling (i.e., sampler did not close completely, etc.);
- Presence or absence of overlying water in sampler;
- Sample depth (i.e., sediment surface to bottom of sampler);
- Description of sediment type (i.e., silt, sand, clay, mud, shells, detritus);
- Description of sample color (i.e., black, brown);
- Description of sample odor, if readily apparent (i.e., sulfur, oily, sewage, none; Note: do not intentionally smell sample to evaluate odor);
- Description of surface biology (i.e., molluscs, crustacea, oligochaetes, midge present); and,

- Sample processing procedure used (i.e., number of grabs taken at site, volume of sample taken from each grab, total volume collected, homogenization methods, sub-sampling methods, type of containers used; Table 3).

Any grab samples that are not intact following retrieval (i.e., low volume, partially washed out, incomplete closure of sampler) will be discarded. The top-layer sediment sample will be obtained by removing approximately 300 mL of sediment from one side of the sampler, taking care to remove only about the top 10 cm of material. This material will be transferred into a stainless steel sample collection container using a stainless steel spoon. This material will then be homogenized and used to fill two glass jars (1- 4oz and 1- 8 oz). One sub-sample will be used for off-site XRF analysis to determine metal concentrations. This sample will be stored at 4°C until dried to a constant weight. Another sample will be stored at 4°C and possibly analyzed later by EPA Method 3052 to confirm the results of the field chemistry results (i.e., at least 10% of these samples will be used to validate the XRF results).

The grab sampler and other equipment will be decontaminated following collection and processing of the sample collected at each sampling station. First, a stainless steel or Teflon spatula will be used to scrape the remaining sediment from the sampler into the waste sediment bucket. Next, the sampler will be scrubbed with a long bristle scrub brush and rinsed with ambient water. Then, the dredge will be squirted with Alconox solution, scrubbed, and rinsed. This decontamination process should also be applied to the stainless steel pan into which the dredge is placed when brought onboard the vessel. The waste sediment that has accumulated in the waste sediment bucket should be returned to the water after all sampling has been completed at the sampling station (i.e., the excess sediment should be returned to the water before leaving the sampling station).

Phase II:

A composite sediment sample will be collected from a located within about a 10-m radius of the designated coordinates at each site; however, the area of the site can be expanded to encompass a larger radius (e.g., 20 m) if required to obtain a suitable sample of fine sediment. The Ekman grab sampler will be used to collect sediment to a depth of about 10 cm. Upon retrieval, the sampler should be placed in the stainless steel tray to avoid contact with any contaminants that may be present on the deck of the boat.

The contents of the Ekman dredge will be emptied into the stainless steel bucket and deployed for additional samples until the buckets has at least 3 liters of sediment. The sediment will be mixed thoroughly and transferred to the 2 liter container. Once the container has been filled and sealed, a GPS reading will be taken from the roughly center of the area within which the material was collected. A description of the sampling area and the positional data will be recorded on the field data collection sheet. Several photographs of the samples and the sampling station will then be taken at this time. The grab sampler and other equipment will be decontaminated as described above following collection and processing of the sample collected at each sampling station.

7.3 Other Precautions to Avoid Sample Contamination

Generation of reliable data on sediment quality conditions is a primary objective of the sampling program. As such, all reasonable efforts should be made to minimize the potential for sample contamination during the sample collection, handling, and processing process. At a minimum, steps that should be taken to avoid sample contamination include:

- Stabilizing the boat by anchoring and shutting off the motor upon arrival at the sampling station;
- Ensuring that sediment samples do not come in contact with any item that has not undergone the approved decontamination process;
- Ensuring that any utensils that are used in the sediment sampling process do not come in contact with any item that has not undergone the approved decontamination process (the sampler and other sampling utensils should be placed in the stainless steel pan during transit between sampling stations);
- Fully decontaminating all sampling equipment after sampling has been completed at a sampling station; and,
- Prohibiting any activity on the sampling vessel that could result in sample contamination (e.g., refueling with sediment samples or sample equipment on board, smoking, consumption of food or drinks during the sampling process; Note: there will be a cooler on deck for food and drinks that are to be consumed at appropriate times).

7.4 Precautions to Avoid Exposure to Contaminated Sediments

It is anticipated that contaminated sediment will be routinely encountered during sampling throughout much of the study area. As such, the sampling crew should take precautions to minimize exposure to potentially toxic and/or bioaccumulative substances. At a minimum, steps that should be taken include:

- Handling sampling equipment and sediment samples carefully;
- Avoiding direct dermal contact with sediments; and,
- Wearing protective equipment, such as gloves, safety glasses, long-sleeved shirts, long pants, rubber boots, and/or rain gear.

More detailed guidance on avoiding hazards during sampling and minimizing the potential for personal injury is provided in the project Health and Safety Plan.

8.0 Sample Handling and Preparation

Phase I:

Procedures for handling and preparing samples for Phase I for chemical analysis should follow the procedures described in ASTM (2008c). Briefly, following collection, sediment samples will be processed on board the sampling vessel. Processing of grab samples will consist of sample homogenization using a stainless steel spoon. Care will be taken to minimize the entrainment of air into the sample during homogenization of sediment samples. Samples will be considered to be homogenized when the entire sediment sample has a uniform texture, color, and consistency.

For the top-layer sediment samples, sub-samples will be obtained for chemical characterization following homogenization. It is anticipated that the following minimum sub-sample volumes will be needed to support the various chemical analyses currently planned for the Phase I sediment samples:

Sediment Chemistry

- XRF analysis for metals	125 mL (4oz)
- ICP-AES analysis for metals	125 mL (4oz)

Following sample homogenization, sub-samples should be transferred to labeled 125 ml (4oz) containers using stainless steel spoons and high-density polyethylene funnels. All containers should be filled to the top to minimize exposure of the sediment to air. After sealing off the containers, the jar should be further secured using electrical tape (i.e., to minimize the potential for sample spillage during shipping). Following sample preparation, the sub-samples should be stored at 4^{o} C until packed for transport/shipping. Samples should be carefully packed with bubble wrap and blue ice, and transferred to the appropriate laboratory for storage in 48 L plastic coolers, along with the appropriate chain of custody forms. If samples are shipped, an inventory must be maintained of all samples that are shipped each day to facilitate confirmation of receipt the following business day.

Phase II:

Briefly, following collection, sediment samples will be processed on board the sampling vessel. Processing of grab samples will consist of sample homogenization using a stainless steel spoon. Care will be taken to minimize the entrainment of air into the sample during homogenization of sediment samples. Samples will be considered to be homogenized when the entire sediment sample has a uniform texture, color, and consistency.

For the top-layer sediment samples, sub-samples will be obtained for toxicity and chemical characterization following homogenization. It is anticipated that the following minimum sub-sample volumes will be needed to support the various chemical analyses currently planned for the samples:

2 liters (1 L for toxicity testing and 1 L for physical and chemical characterization of each sediment sample)

Following sample homogenization, samples should be transferred to labeled 2 L containers using stainless steel spoons and high-density polyethylene funnels. All containers should be filled to the top to minimize exposure of the sediment to air. After sealing off the containers, the jar should be further secured using electrical tape (i.e., to minimize the potential for sample spillage during shipping). Following sample preparation, the sub-samples should be stored at 4^{o}C until packed for transport. Samples should be placed in coolers with ice or in a refrigeration truck for transport to the CERC in Columbia MO by October 24, 2008.

9.0 Chemicals of Potential Concern

Studies of the upstream watersheds of the Tri-State District have identified cadmium, lead, and zinc as the primary constituents that are elevated due to mining activity.[3] Along with their prevalence throughout the Tri-State area, the adverse biological effects of these metals are well documented.[4] In order to identify potential injury from mining activity, sediment samples will be analyzed for these target metals. The list of priority analytes and associated data quality objectives for the chemical analyses are presented in Table 2.

10.0 Roles and Responsibilities of Sampling Team

Samples to support the sediment quality investigation will be systematically collected within the sampling area. The sampling teams will each include three individuals who will be responsible for operating the sampling vessel, collecting and preparing sediment samples, conducting on-site analyses of sediment samples, and preparing and shipping samples for possible future analysis. All members of the sampling crew will be required to wear personal flotation devices at all times while on the water. Quality assurance and control (QA/QC) for the field portion of this investigation will be directed by Suzanne Dudding and John Miesner of the US FWS.

The sample collection crew will be responsible for ensuring that all necessary sampling equipment and associated supplies are loaded onto the sampling vessel(s) each day, verifying the locations of the stations that are sampled (using handheld GPS), collecting sufficient volumes of sediments to support analyses of sediment chemistry, preparing and labeling sediment samples, decontaminating the sediment samplers between deployments at a site, and following the completion of sampling activities at each station.

Sediment samples should be cooled to 4^{o}C and transported in a manner that assures that this temperature is maintained. Any sub-samples that are lost or damaged during transport must be identified by sampling station and recorded. If a commercial shipping service is used, sub-samples may be shipped on Monday, Tuesday, Wednesday, Thursday, and Sunday only (to avoid weekend delivery to laboratories). Any sub-samples that are not shipped on the date of collection must be held on site at 4^{o}C and shipped on the next appropriate shipping day (i.e., on ice in

[3] The adverse effects of mining in the Tri-State District have been documented in several sources including, but not limited to: Dames and Moore, 1993 and 1995; Ferrington et al, 1995; USGS, 2005.
[4] Eisler, 2000.

coolers). Unused portions of sediment samples should be disposed of at the station that the samples were collected.

Sample collection and disposition will be clearly documented. Individual sampling containers s should be labeled with the sample identification number as presented in Table 2, with pre-printed labels. At each sampling station, the data collection form will be filled out (see example in Addendum 1A). Samples will then be stored in coolers on ice. Samples transported to a laboratory will be packaged in a cooler with blue ice as described in Section 8.0 and a chain of custody (COC) manifest will be prepared (see example in Addendum 2B). A copy of the COC will be maintained with the sample records, and the initial disposition will be noted on the data collection form. The laboratory will also return a copy of the updated COC to the sender upon receipt and acceptance.

11.0 Quality Assurance

Phase I:

Generation of good quality sediment chemistry data is essential for supporting the 2008 sediment quality investigation of Grand Lake. To avoid problems associated with data reliability, it is necessary to implement adequate quality assurance measures in the sampling program, during data collection and analysis. In this study, the quality of data analyzed by portable XRF will be evaluated by conducting laboratory analysis of a sub-set (minimum of 10%) of the field-collected samples using methods consistent with those used in previous sediment investigations in the Tri-State area. XRF analysis of all 96 samples will be performed using EPA Method 6200 by FWS personnel after completion of the field collection. Samples for XRF analysis will be dried to a constant weight at the FWS field office in Tulsa, OK and analyzed as whole sediments at the FWS field office in Manhattan, KS. John Miesner will direct quality assurance and control during XRF analysis.

The samples that are selected for confirmatory chemical analysis will be identified following review and evaluation of the XRF data. Samples sent to the laboratory for confirmatory analysis will be sieved to 64 µm. Chemical analysis of metal content will be performed on the finer, sieved sediment.[5] Sieved samples will then be digested using EPA Method 3052 (Microwave assisted acid digestion of siliceous and organically based matrices) and analyzed by ICP-AES using EPA Method 6010B. Standard methods for XRF and laboratory analysis are appended to this document.

Phase II:

Sediment chemistry measures on the 40 Phase II sediment samples to be performed under the direction of CERC will include pore-water metals, simultaneously-extracted metals (SEM), acid-volatile sulfide (AVS), total organic carbon (TOC), and particle size distribution (Table 1; all of these analyses will be performed by the Patuxent Analytical

[5] Note that this methodology is in line with EPA methodology for previous sediment sampling within the Tri-State area. Samples to be analyzed using XRF methodology will not be sieved, because the range of error of this device would not indicate a difference between sieved and un-sieved sediments (pers. comm., Miesner).

Control Facility [PACF] except for pore-water metals which will be analyzed by CERC). USFWS will measure total metal concentrations in these 40 Phase II sediment samples using XRF. CERC (through the PACF) will also provide measures of total recoverable metals in about 10% of the Phase I sediment samples to confirm relationships between XRF measures and acid-extracted metals measured using inductively coupled plasma atomic emission spectrophotometry (ICPAES; EPA Method 3051A). These selected measures of sediment toxicity and sediment chemistry (total metals, SEM, and AVS) have been identified as sensitive indicators of metal bioavailability in contaminated sediments of the TSMD (MacDonald et al. 2008). Information from this study will be combined with existing sediment chemistry data for Grand Lake to evaluate relationships between sediment toxicity and sediment chemistry in Grand Lake sediments and to determine spatial extent of metals contamination in sediment in Grand Lake.

12.0 Examples of Forms for Sampling Program

Examples of forms that will be used in the sample program are presented as addenda, including the data collection form (Addendum 1A) and chain of custody form (Addendum 1B).

13.0 References

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Table 1 - GPS locations and sample designations for sediment sampling – Phase I

Sample Site	X	Y
Transect 1		
2008_GL_T1_01	-95.0415	36.4699
2008_GL_T1_02	-95.0391	36.4711
2008_GL_T1_03	-95.0367	36.4721
2008_GL_T1_04	-95.0340	36.4733
2008_GL_T1_05	-95.0318	36.4743
2008_GL_T1_06	-95.0298	36.4753
2008_GL_T1_07	-95.0277	36.4762
2008_GL_T1_08	-95.0262	36.4769
Transect 2		
2008_GL_T2_01	-95.0172	36.5008
2008_GL_T2_07	-95.0170	36.4896
2008_GL_T2_06	-95.0171	36.4915
2008_GL_T2_05	-95.0171	36.4934
2008_GL_T2_02	-95.0172	36.4991
2008_GL_T2_03	-95.0172	36.4974
2008_GL_T2_04	-95.0171	36.4954
2008_GL_T2_08	-95.0170	36.4882
Transect 3		
2008_GL_T3_01	-94.9739	36.5089
2008_GL_T3_02	-94.9732	36.5081
2008_GL_T3_03	-94.9724	36.5071
2008_GL_T3_04	-94.9715	36.5060
2008_GL_T3_05	-94.9706	36.5050
2008_GL_T3_06	-94.9697	36.5038
2008_GL_T3_07	-94.9687	36.5028
2008_GL_T3_08	-94.9678	36.5017
Transect 4		
2008_GL_T4_01	-94.9579	36.5379
2008_GL_T4_02	-94.9563	36.5370
2008_GL_T4_03	-94.9547	36.5361
2008_GL_T4_04	-94.9529	36.5350
2008_GL_T4_05	-94.9510	36.5341
2008_GL_T4_06	-94.9491	36.5328
2008_GL_T4_07	-94.9470	36.5317
2008_GL_T4_08	-94.9452	36.5305
Transect 5		
2008_GL_T5_01	-94.9133	36.5528
2008_GL_T5_02	-94.9122	36.5513
2008_GL_T5_03	-94.9110	36.5499
2008_GL_T5_04	-94.9097	36.5483
2008_GL_T5_05	-94.9085	36.5469
2008_GL_T5_06	-94.9075	36.5456
2008_GL_T5_07	-94.9065	36.5443
2008_GL_T5_08	-94.9054	36.5430

Sample Site	X	Y
Transect 7		
2008_GL_T7_01	-94.8455	36.5743
2008_GL_T7_02	-94.8440	36.5751
2008_GL_T7_03	-94.8424	36.5763
2008_GL_T7_04	-94.8408	36.5772
2008_GL_T7_05	-94.8393	36.5782
2008_GL_T7_06	-94.8378	36.5792
2008_GL_T7_07	-94.8365	36.5801
2008_GL_T7_08	-94.8352	36.5808
Transect 8		
2008_GL_T8_01	-94.8479	36.6253
2008_GL_T8_02	-94.8467	36.6227
2008_GL_T8_03	-94.8452	36.6196
2008_GL_T8_04	-94.8436	36.6162
2008_GL_T8_05	-94.8422	36.6131
2008_GL_T8_06	-94.8406	36.6096
2008_GL_T8_07	-94.8388	36.6063
2008_GL_T8_08	-94.8373	36.6030
Transect 9		
2008_GL_T9_01	-94.8091	36.6493
2008_GL_T9_02	-94.8091	36.6474
2008_GL_T9_03	-94.8091	36.6455
2008_GL_T9_04	-94.8091	36.6434
2008_GL_T9_05	-94.8091	36.6415
2008_GL_T9_06	-94.8090	36.6394
2008_GL_T9_07	-94.8090	36.6374
2008_GL_T9_08	-94.8090	36.6356
Transect 10		
2008_GL_T10_01	-94.7775	36.6522
2008_GL_T10_02	-94.7764	36.6511
2008_GL_T10_03	-94.7752	36.6501
2008_GL_T10_04	-94.7737	36.6487
2008_GL_T10_05	-94.7724	36.6475
2008_GL_T10_06	-94.7708	36.6460
2008_GL_T10_07	-94.7696	36.6447
2008_GL_T10_08	-94.7683	36.6434
Transect 11		
2008_GL_T11_01	-94.7792	36.7302
2008_GL_T11_02	-94.7783	36.7301
2008_GL_T11_03	-94.7773	36.7299
2008_GL_T11_04	-94.7761	36.7298
2008_GL_T11_05	-94.7748	36.7295
2008_GL_T11_06	-94.7735	36.7292
2008_GL_T11_07	-94.7723	36.7290
2008_GL_T11_08	-94.7713	36.7287

Sample Site	X	Y
Transect 6		
2008_GL_T6_01	-94.8467	36.5596
2008_GL_T6_02	-94.8477	36.5586
2008_GL_T6_03	-94.8489	36.5571
2008_GL_T6_04	-94.8502	36.5558
2008_GL_T6_05	-94.8516	36.5544
2008_GL_T6_06	-94.8531	36.5528
2008_GL_T6_07	-94.8545	36.5514
2008_GL_T6_08	-94.8560	36.5500

Sample Site	X	Y
Transect 12		
2008_GL_T12_01	-94.7548	36.7946
2008_GL_T12_02	-94.7541	36.7948
2008_GL_T12_03	-94.7535	36.7950
2008_GL_T12_04	-94.7528	36.7951
2008_GL_T12_05	-94.7521	36.7953
2008_GL_T12_06	-94.7515	36.7955
2008_GL_T12_07	-94.7508	36.7957
2008_GL_T12_08	-94.7502	36.7959

Table 2 Target analytes for sediment samples 2008 sediment quality assessment of Grand Lake – Phase I

Sample Id Number	Total Metals by XRF	Cd by ICP-AES	Pb by ICP-AES	Zn by ICP-AES
Grand Lake Transect 1				
2008_GL_T1_01	x	x	x	x
2008_GL_T1_02	x			
2008_GL_T1_03	x			
2008_GL_T1_04	x			
2008_GL_T1_05	x			
2008_GL_T1_06	x			
2008_GL_T1_07	x			
2008_GL_T1_08	x	x	x	x
Grand Lake - Transect 2				
2008_GL_T2_01	x			
2008_GL_T2_07	x			
2008_GL_T2_06	x			
2008_GL_T2_05	x	x	x	x
2008_GL_T2_02	x			
2008_GL_T2_03	x			
2008_GL_T2_04	x			
2008_GL_T2_08	x			
Grand Lake - Transect 3				
2008_GL_T3_01	x	x	x	x
2008_GL_T3_02	x			
2008_GL_T3_03	x			
2008_GL_T3_04	x			
2008_GL_T3_05	x			
2008_GL_T3_06	x			
2008_GL_T3_07	x			
2008_GL_T3_08	x			
Grand Lake - Transect 4				
2008_GL_T4_01	x			
2008_GL_T4_02	x			
2008_GL_T4_03	x			
2008_GL_T4_04	x			
2008_GL_T4_05	x			
2008_GL_T4_06	x	x	x	x
2008_GL_T4_07	x			
2008_GL_T4_08	x			
Grand Lake - Transect 5				
2008_GL_T5_01	x			
2008_GL_T5_02	x			

Sample Id Number	Total Metals by XRF	Cd by ICP-AES	Pb by ICP-AES	Zn by ICP-AES
Grand Lake - Transect 7				
2008_GL_T7_01	x			
2008_GL_T7_02	x			
2008_GL_T7_03	x			
2008_GL_T7_04	x			
2008_GL_T7_05	x			
2008_GL_T7_06	x			
2008_GL_T7_07	x			
2008_GL_T7_08	x	x	x	x
Grand Lake - Transect 8				
2008_GL_T8_01	x			
2008_GL_T8_02	x			
2008_GL_T8_03	x			
2008_GL_T8_04	x			
2008_GL_T8_05	x	x	x	x
2008_GL_T8_06	x			
2008_GL_T8_07	x			
2008_GL_T8_08	x			
Grand Lake - Transect 9				
2008_GL_T9_01	x	x	x	x
2008_GL_T9_02	x			
2008_GL_T9_03	x			
2008_GL_T9_04	x			
2008_GL_T9_05	x			
2008_GL_T9_06	x			
2008_GL_T9_07	x			
2008_GL_T9_08	x			
Grand Lake - Transect 10				
2008_GL_T10_01	x			
2008_GL_T10_02	x			
2008_GL_T10_03	x	x	x	x
2008_GL_T10_04	x			
2008_GL_T10_05	x			
2008_GL_T10_06	x			
2008_GL_T10_07	x			
2008_GL_T10_08	x			
Grand Lake - Transect 11				
2008_GL_T11_01	x			
2008_GL_T11_02	x			

2008_GL_T5_03	x				2008_GL_T11_03	x			
2008_GL_T5_04	x				2008_GL_T11_04	x	x	x	x
2008_GL_T5_05	x				2008_GL_T11_05	x			
2008_GL_T5_06	x				2008_GL_T11_06	x			
2008_GL_T5_07	x	x	x	x	2008_GL_T11_07	x			
2008_GL_T5_08	x				2008_GL_T11_08	x			
Grand Lake - Transect 6					***Grand Lake - Transect 12***				
2008_GL_T6_01	x				2008_GL_T12_01	x			
2008_GL_T6_02	x	x	x	x	2008_GL_T12_02	x			
2008_GL_T6_03	x				2008_GL_T12_03	x			
2008_GL_T6_04	x				2008_GL_T12_04	x	x	x	x
2008_GL_T6_05	x				2008_GL_T12_05	x			
2008_GL_T6_06	x				2008_GL_T12_06	x			
2008_GL_T6_07	x				2008_GL_T12_07	x			
2008_GL_T6_08	x	x	x	x	2008_GL_T12_08	x	x	x	x

Table 3. Sampling equipment required to support the 2008 investigation of sediment quality conditions in Grand Lake

Cleaning supplies – Phase I & II

- Alconox solution - 4-L
- Deionized/reverse osmosis water - 4 L
- Polyethylene squeeze bottles or spray bottles - 3 x 1 L
- Long bristle scrub brush - 3

Sampling supplies – Phase I & II

- Maps of the study area - 3
- Sharpie pens - 5 to 10
- Ekman grab sampler - 3
- Rope for deploying sampler (>50' long)
- Stainless steel spoons - 3
- Stainless steel pan (approximately 18" x 12" x 4")
- Sample collection forms - 96
- Labels for whirl-paks (or mark directly with Sharpie)
- Latex gloves - non-powdered - 2 boxes of 50
- Siphon or turkey baster for removing excess water from sampler (polyethylene)
- Stainless steel homogenization buckets - 3 x 1 L; 3 x 5 L
- Sediment waste bucket - 3 x 25 L
- Polyethylene funnels - 3 (sized to fit into mouth of whirl-pak bags)
- Wildco buckets - 3

Measurement supplies Phase I & II

- GPS (handheld)
- Sonar or other depth measure

Shipping and storage supplies – Phase I

- 48-L plastic coolers - 6
- FedEx labels for shipments - as needed
- Paper towels - 6 rolls
- Blue ice to maintain samples at $4^{o}C$ - 6 packs
- Shipping manifests - as needed
- Electrical tape - 6 rolls

Personal supplies Phase I & II

- Personal Flotation Device for each member of the sampling crew
- Gloves
- Rubber boots
- Cooler for onsite food and beverage consumption - 6 x 48 L
- Long pants
- Rain/ foul-weather gear

Miscellaneous supplies Phase I & II

- Forms for data collection on samples - 96
- Sampling vessel
- Sample book (write in rain)
- Digital camera with accessories (spare batteries; extra memory chips)
- Boat anchors - 3

Table 4 Volume, container material, preservation specifications, and holding times for Grand Lake.2008 - Phase I

Parameter Analyzed	Laboratory[1]	Approximate Volume	Container Material	Preservation Method	Holding Time
Metals (by XRF)	FWS facility	125 mL	4 oz glass jar	4°C until dried, then room temperature	28-d
Metals (by EPA Method 6010B)	FWS laboratory	125 mL	4 oz glass jar	4°C	14-d

d = day; XRF = X-Ray fluorescence

Table 5. Test conditions for conducting long-term sediment toxicity tests with the amphipod *Hyalella azteca* with Grand Lake sediments (adapted from Table A6.1 in ASTM 2008a [analogous tables are included in USEPA 2000]).

Parameter	Conditions
1. Test type	Whole-sediment toxicity test with renewal of overlying water conducted with 40 field-collected sediment samples from Grand Lake and with West Bearskin sediment as a control sediment (about 3% total organic carbon; Ingersoll et al. 1998)
2. Temperature	23 ± 1°C
3. Light quality	Wide-spectrum fluorescent lights
4. Illuminance	About 200 lux
5. Photoperiod	16L:8D
6. Test chamber	300-mL high-form lipless beaker
7. Sediment volume	100 mL (sediment added to exposure beakers on Day -7 and held under static conditions at 23°C until Day -1)
8. Overlying water volume	175 ml
9. Renewal of overlying water	2 volume additions/d
10. Age of organisms	About 7-d-old organisms (archive 20 amphipods on Day 0 for length measurement)
11. Number of organisms/chamber	10
12. Number of replicate chambers/treatment	Toxicity testing: 4/treatment for each Grand Lake sediment treatment and 8/control treatment. Chemistry: 1 (for sampling pore-water metals with peeper samples during from Day 0 to Day 7 with amphipods and food also added to each chamber) Total number of replicates/treatment: 5 Volume of sediment/treatment: 0.1 L/chamber x 5 chambers = 0.5 L Total number of chambers: 5x41=205+4=209 Total number of exposure systems: 209/192 =1.1
13. Feeding	Yeast-cerophyl-trout chow (YCT), fed 1.0 mL (1800 mg/L stock) daily to each test chamber
14. Aeration	None, unless dissolved oxygen in overlying water drops below 2.5 mg/L
15. Overlying water	Well water diluted with deionized water to a hardness of about 100 mg/L (as $CaCO_3$), alkalinity 85 mg/L (as $CaCO_3$), and pH about 7.8.
16. Test chamber cleaning	If screens become clogged during a test; gently brush the outside of the screen
17. Overlying water quality	Hardness, alkalinity, conductivity, and ammonia at the beginning and end of a test and on the days that growth is subsampled. Temperature daily. Dissolved oxygen (DO) and pH three times/week. Concentrations of DO should be measured more often if DO has declined by more

Parameter	Conditions
	than 1 mg/L since previous measurement.
18. Chemistry sampling	Pore-water quality: Day -7 by centrifugation at 4ºC for 15 minutes at 5,200 rpm; 0.5 L whole-sediment/treatment. Hardness, alkalinity, conductivity, ammonia, pH (in accordance with methods outlined in Ingersoll et al. 2008). Pore Pore-water metals: Day 0 to Day 7 sampled with peepers (in accordance with methods outlined in Ingersoll et al. 2008). Whole sediment: CERC will provide measures of simultaneously-extracted metals (SEM), acid-volatile sulfide (AVS), total organic carbon (TOC), and particle size distribution in the 40 Phase I sediment samples (through the PACF in basic accordance with methods outlined in Ingersoll et al. 2008). USFWS will measure total metal concentrations in these Phase I sediment samples using XRF. CERC (through the PACF) will also provide measures of total recoverable metals in about 10% of the Phase I sediment samples to confirm relationships between XRF measures and acid-extracted metals measured using inductively coupled plasma atomic emission spectrophotometry (ICPAES; EPA Method 3051A).
19. Endpoints	28-d survival and growth (length, weight, total biomass; in accordance with methods outlined in Ingersoll et al. 2008)
20. Test acceptability	Minimum mean control survival of 80% on Day 28. Additional performance-based criteria specifications are outlined in Table 7).

Table 6. General activity schedule for conducting a long-term sediment toxicity test with *Hyalella azteca* (adapted from Table A6.2 in ASTM 2008a, an analogous table is included in USEPA 2000. NOTE: THE TEST WILL BE CONDUCTED FOR 28- MONITORING SURVIVAL AND GROWTH OF AMPHIPODS.

Day	Activity
Pre-Test	
-7	Place sediment in exposure beakers under static conditions.
–1	Isolate amphipods from culture and feed and observe isolated amphipods, monitor water quality. Start renewing overlying water.
Sediment Test	
0	Measure total water quality (pH, temperature, dissolved oxygen, hardness, alkalinity, conductivity, ammonia). Transfer ten 7- to 8-day old amphipods into each test chamber. Release organisms under the surface of the water. Add 1.0 mL of YCT (1800 mg/L stock) into each test chamber. Archive 20 test organisms for length determination or archive 80 amphipods for dry weight determination. Observe behavior of test organisms.
1 to 27	Add 1.0 mL of YCT to each test beaker. Measure temperature daily, conductivity weekly, and dissolved oxygen (DO) and pH three times/week. Observe behavior of test organisms.
28	Measure temperature, dissolved oxygen, pH, hardness, alkalinity, conductivity and ammonia. End the sediment-exposure portion of the test by collecting the amphipods with a #40 mesh sieve (425-µm mesh; U.S. standard size sieve). Use four replicates for growth measurements: count survivors and preserve organisms in sugar formalin for growth measurements. Eight replicates for reproduction measurements: Place survivors in individual replicate water-only beakers and add 1.0 mL of YCT to each test beaker/d and 2 volume additions/d of overlying water.
Reproduction Phase	NOTE: THE TEST WILL END ON DAY 28 WITH NO MEASURE OF REPRODUCTION.
29 to 35	Feed daily. Measure temperature daily, conductivity weekly, DO and pH three times a week. Measure hardness and alkalinity weekly. Observe behavior of test organisms.
35	Record the number of surviving adults and remove offspring. Return adults to their original individual beakers and add food.
36 to 41	Feed daily. Measure temperature daily, conductivity weekly, DO and pH three times a week. Measure hardness and alkalinity weekly. Observe behavior of test organisms.
41	Measure total water quality (pH, temperature, dissolved oxygen, hardness, alkalinity, conductivity, ammonia).
42	Record the number of surviving adults and offspring. Surviving adult amphipods on Day 42 are preserved in sugar formalin solution. The number of adult males in each beaker is determined from this archived sample. This information is used to calculate the number of young produced per female per replicate from Day 28 to Day 42.

Table 7. Test acceptability requirements for conducting a long-term sediment toxicity test with *Hyalella azteca* (adapted from Table A6.1 in ASTM 2008a, an analogous table is included in USEPA 2000).

A. It is recommended for conducting the 42-day test with *H. azteca* that the following performance criteria be met:

1. Age of *H. azteca* at the start of the test should be 7- to 8-day old. Starting a test with substantially younger or older organisms may compromise the reproductive endpoint.
2. Average survival of *H. azteca* in the control sediment on Day 28 should be greater than or equal to 80%.
3. Laboratories participating in round-robin testing (Section 17.6 of ASTM 2008a) reported after 28-day sediment exposures in a control sediment (West Bearskin), survival >80% for >88% of the laboratories; length >3.2 mm/individual for >71% of the laboratories; and dry weight >0.15 mg/individual for 66% of the laboratories. Reproduction from Day 28 to Day 42 was >2 young/female for 71% of the laboratories participating in the round-robin testing. Reproduction was more variable within and among laboratories; hence, more replicates might be needed to establish statistical differences among treatments with this endpoint.
4. Hardness, alkalinity, and ammonia in the overlying water typically should not vary by more than 50% during the sediment exposure, and dissolved oxygen should be maintained above 2.5 mg/L in the overlying water.

B. Performance-based criteria for culturing *H. azteca* include the following:

1. It may be desirable for laboratories to periodically perform 96-h water-only reference-toxicity tests to assess the sensitivity of culture organisms (Section 11.16.2 of ASTM 2008a). Data from these reference toxicity tests could be used to assess genetic strain or life-stage sensitivity of test organisms to select chemicals.
2. Laboratories should track parental survival in the cultures and record this information using control charts if known-age cultures are maintained. Records should also be kept on the frequency of restarting cultures and the age of brood organisms.
3. Laboratories should record the following water-quality characteristics of the cultures at least quarterly: pH, hardness, alkalinity, and ammonia. Dissolved oxygen in the cultures should be measured weekly. Temperature in the cultures should be recorded daily. If static cultures are used, it may be desirable to measure water quality more frequently.
4. Laboratories should characterize and monitor background contamination and nutrient quality of food if problems are observed in culturing or testing organisms.
5. Physiological measurements such as lipid content might provide useful information regarding the health of the cultures.

C. Additional requirements:

1. All organisms in a test must be from the same source.
2. Storage of sediments collected from the field should follow guidance outlined in Section 10.2 of ASTM (2008a).
3. All test chambers (and compartments) should be identical and should contain the same amount of sediment and overlying water.
4. Negative-control sediment and appropriate solvent controls must be included in a test. The concentration of solvent used must not adversely affect test organisms.
5. Test organisms must be cultured and tested at 23°C (±1 °C).

6. The mean of the daily test temperature must be within ± 1°C of 23°C. The instantaneous temperature must always be within ±3°C of 23°C.

7. Natural physico-chemical characteristics of test sediment collected from the field should be within the tolerance limits of the test organisms.

Table 8. Summary of test conditions for conducting reference toxicant tests (in basic accordance with ASTM 2008a,b and USEPA 2000).

Test chemical:	Sodium chloride (NaCl)
Test type:	Static
Test Duration:	48 h
Temperature:	23°C
Light quality:	Ambient laboratory light
Light intensity:	200 lux
Photoperiod:	16L:8D
Test chamber size:	50 ml
Test solution volume:	30 ml
Renewal of solution:	None
Age of test organism:	about 7-d old
No. organisms per test chamber:	5
No. replicate chambers per concentration:	4
Feeding:	No feeding
Chamber cleaning:	None
Aeration:	None
Dilution water:	ASTM reconstituted hard water (170 mg/L as $CaCO_3$; ASTM 2008b)
Dilution factor:	0.5
Test concentration:	0, 0.5, 1, 2, 4, and 8 g NaCl/L
Chemical residues:	Salinity in each NaCl solution will be measured at the beginning and the end of test
Water quality:	Dissolved oxygen, pH, conductivity, hardness, and alkalinity will be determined at the control, medium, and high NaCl concentrations at the beginning and the end of test
Endpoint:	Survival
Test acceptability criterion:	≥90% control survival